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13a. Description of Change This ECN has been generated in order to update the document to reflect results of recent data/information evaluation. Replace pages: ES-3 through ES-6, 3-1, 3-2, 5-11 through 5-16, 6-1, 6-2, 7-3, and 7-4							
13b. Design Baseline Document? <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No							
14a. Justification (mark one) Criteria Change <input checked="" type="checkbox"/> Design Improvement <input type="checkbox"/> Environmental <input type="checkbox"/> Facility Deactivation <input type="checkbox"/> As-Found <input type="checkbox"/> Facilitate Const <input type="checkbox"/> Const. Error/Omission <input type="checkbox"/> Design Error/Omission <input type="checkbox"/>							
14b. Justification Details A tank characterization report page change revision is required to reflect the results of recent evaluation of data/information pertaining to adequacy of tank sampling for safety screening purposes (Reynolds et al. 1999, Evaluation of Tank Data for Safety Screening, HNF-4217, Rev. 0, Lockheed Martin Hanford Corporation, Richland, Washington).							
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Tank Characterization Report for Double-Shell Tank 241-SY-103

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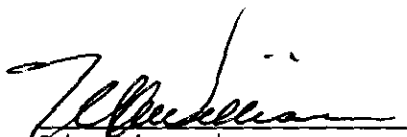
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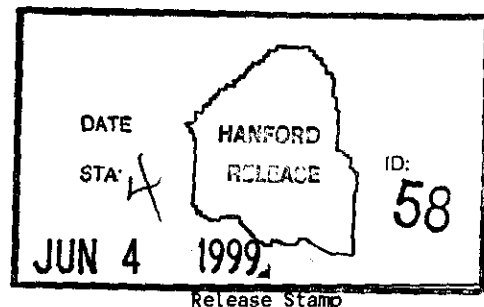
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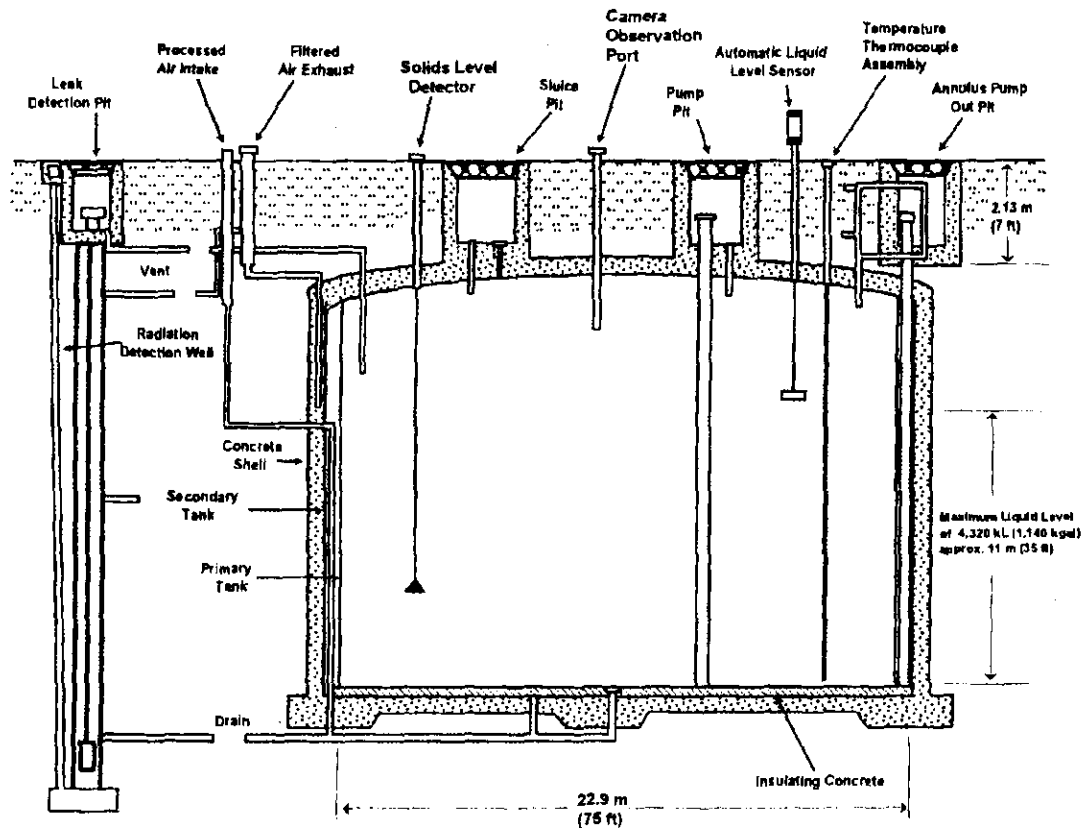

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Figure ES-1. Basic Design of a Double-Shell Tank.



Not to Scale

This report summarizes three sampling and analysis events.

- The solids and supernate compositions are based on the core sample taken in 1994.
- The crust was evaluated for safety concerns using auger solids in 1994.
- The physical properties of the solids presented were taken from 1986 and 1994 core segment samples.

An unreviewed safety question raised concern that the crust of the tank waste could become sufficiently hot during core sampling activities to initiate an exothermic reaction or ignite hydrogen gas, if present (Johnson 1994). General safety screening analyses were performed on the crust prior to core sampling in response to the unreviewed safety question. The differential scanning calorimetry (DSC) results did not exhibit exotherms, thus indicating that it was safe to obtain a push-mode core sample (Schreiber 1995).

Two data quality objectives (DQOs) were applicable to the 1994 core sampling event: the *Flammable Gas Tank Safety Programs: Data Requirements for Core Sample Analysis Developed Through the Data Quality Objective Process* (McDuffie and Johnson 1994) and the *Tank Safety Screening Data Quality Objective* (Babad and Redus 1994). The flammable gas safety DQO requires one core, and the safety screening DQO requires two cores taken from two widely separated risers. Because of safety concerns, only one core was acquired; therefore, although the objectives were met for the flammable gas safety DQO, they were not met originally for the safety screening DQO. Safety screening analyses were performed on the one core obtained. A recent evaluation of sampling and analytical data concludes that

although only one core was recovered and analyzed, the data for tank 241-SY-103 are sufficient to address the safety screening DQO criteria (Reynolds et al. 1999). The safety screening issue is now considered closed for this tank.

Safety screening analyses were performed to evaluate the potential for exothermic reactions in the waste, criticality, and tank vapor flammability. The DSC results for one drainable liquid (segment 9, 559 J/g) and one lower semi-segment (segment 13, 630 J/g) exceeded the safety screening exothermic enthalpy criteria of 480 J/g based on the dry weight of the sample.¹ Most segments exhibited an exotherm, thus indicating that fuel is present throughout the tank.

The exothermic behavior is most likely the result of the reaction of organic complexants with nitrates/nitrites at elevated temperatures. Total organic carbon (TOC) concentrations are relatively high in each segment. The samples with the larger exotherms had dry weight TOC concentrations near 2 weight percent, which is below the safety screening criteria of 3 weight percent. Energy estimates calculated from the TOC concentration, assuming that the TOC is acetate, were all greater than the observed exotherms from DSC analysis except for two samples. Only small amounts of cyanide were found in the waste and do not contribute significantly to the observed energetics.

¹The safety screening criteria at the time of the analysis was 523 J/g, but it has been changed to 480 J/g in later DQOs.

The heat generated by radioactivity in the tank is estimated to be 5,880 W (20,100 Btu/hr), which is well below the criteria (11,720 W [40,000 Btu/hr]) distinguishing a high-heat tank from a low-heat tank. In 1994, tank 241-SY-103 had maximum temperatures ranging from 36 to 39 °C (96 to 103 °F). The trend of the temperature data indicates the waste is cooling.

Total alpha results indicate that the tank is well below the criticality safety criterion of 41 $\mu\text{Ci/g}$, but actinide levels in the solids exceed the transuranic classification of 100 nCi/g. Isotopic analyses indicates that most of the alpha activity is from ^{241}Am and not $^{239/240}\text{Pu}$. The actinide levels in the supernate are well below the transuranic classification.

A standard hydrogen monitoring system was installed in June 1994. Headspace sampling indicates the presence and periodic buildup of hydrogen gas. The largest hydrogen concentration measured was 0.294 volume percent on May 2, 1995. This result is equivalent to 7.4 percent of the lower flammability limit (LFL) and does not exceed the tank safety DQO criterion of 25 percent of the LFL (Babad and Redus 1994).

Ammonia monitors on the SY tank farm indicate the highest ammonia concentration during a gas release event is about 486 ppmv or 0.3 percent of the LFL for ammonia. Additional waste characterization data were obtained to clarify mechanisms for gas generation, retention, and release. These data will be used in models of waste behavior to support evaluation of gas accumulation and development of any needed mitigation methods. Discussion of these mechanisms, models, and mitigation efforts is beyond the scope of this

3.0 TANK SAMPLING OVERVIEW

This section describes three sampling events associated with tank 241-SY-103. A push-mode core was acquired in August and September of 1994 in support of the *Tank Safety Screening Data Quality Objective* (DQO) (Babad and Redus 1994), the *Flammable Gas Tank Safety Program: Data Requirements for Core Sample Analysis Developed Through the Data Quality Objective Process* (McDuffie and Johnson 1994), and *Tank 241-SY-103 Tank Characterization Plan* (Schreiber 1994b and 1995). In June 1994, auger sampling and analysis of the tank's crust material were completed to ensure that further core sampling could be carried out in a safe manner. In 1986, core samples were taken from tank 241-SY-103 in support of retrieval, transport, and pretreatment characterization activities.

Results for the 1994 push-mode core sampling event may be found in *45-Day Safety Screen Results for Tank 241-SY-103, Core 62* and *216-Day Final Report for Tank 241-SY-103 Push Mode, Core 62* reports (Rice 1994 and 1995). The results for the auger sampling event are given in the *45-Day Deliverable for Tank 241-SY-103* (Kocher 1994) and the *136-Day Deliverable for Tank 241-SY-103 Auger Samples, Risers 7A, 14B, and 22A* (Bell 1994). The results for the 1986 sampling event are reported in *Tank 103-SY Dissolution Study - Results of Physical Measurements* (Prignano 1988a), *Tank 103-SY Dissolution Study - Results of Chemical Analyses* (Prignano 1988b), and *Characterization of Waste from Double-Shell Tank 103-SY, A Letter Report for Rockwell Hanford Operations* (Fow et al. 1986). Pre-May 1989 data may not be acceptable for waste decisions because adequate quality control information for the data is not available to assess data quality and enable confident decisions.

3.1 DESCRIPTION OF THE 1994 CORE SAMPLING EVENT

During August and September of 1994, one push-mode core was obtained from riser 14A of tank 241-SY-103. The core consisted of 15 segments and was numbered core 62. A solution of 0.3 molar (*M*) lithium bromide was used for the hydrostatic head fluid (HHF). Originally, a second core sample was planned, but it was not acquired because of safety concerns related to sample pressurization of the last core segment. Because the second core was not acquired, the duplicate sampler requirements of the tank safety screening DQO were not met for this tank (Babad and Redus 1994). However, a recent evaluation has determined that the sampling and analytical data are sufficient to address the safety screening issue for this tank (Reynolds et al. 1999).

Table 3-1 summarizes the sampling information for this event. The third column lists the approximate elevation of the top of each segment as measured from the bottom of the tank, using the solids level measurement of 6.86 m (22.5 ft) taken by manual tape in July 1994 (Schreiber 1994b). It should be noted that the first segment was only to a depth of 10 cm (4 in.). The depth information is given as a guide and is not precise. Table 3-1 also

Table 3-1. Tank 241-SY-103 Core 62 Sampling Information¹.

Segment Number	Customer Sample Number	Sample Elevation ² (cm)	Sample Date ³	Date Received by 222-S Laboratory ³	Extrusion Date ³	Drill String Dose Rate (R/hr)
Riser 14A						
1	94-005	714	8/19/94	8/22/94	8/24/94	2
2	94-006	666	8/19/94	8/22/94	8/24/94	2.2
3	94-007	617	8/23/94	8/25/94	8/26/94	2.2
4	94-008	569	8/23/94	8/25/94	8/26/94	2
5	94-009	521	9/8/94	9/9/94	9/12/94	2.2
6	94-010	473	9/13/94	9/15/94	9/16/94	2.5
7	94-011	425	9/13/94	9/15/94	9/16/94	2
8	94-012	376	9/13/94	9/15/94	9/19/94	2
9	94-013	328	9/13/94	9/15/94	9/19/94	1.9
10	94-014	280	9/13/94	9/15/94	9/20/94	1.7
11	94-015	232	9/16/94	9/19/94	9/21/94	1.9
12	94-016	183	9/16/94	9/19/94	9/22/94	1.7
13	94-017	135	9/16/94	9/19/94	9/23/94	1.8
14	94-018	87	9/19/94	9/21/94	9/23/94	1.8
15	94-019	39	9/19/94	9/21/94	9/26/94	1.5
field blank ⁴	n/a	n/a	n/a	n/a	n/a	n/a
HHF		n/a			n/a	n/a

Notes:

n/a = not available

¹Rice (1994)²As measured from the bottom of the tank to the top of the core segment; values are approximate.³Dates are listed in the mm/dd/yy format.⁴The 222-S Laboratory has no record of receipt or analysis of a field blank.

5.5 EVALUATION OF PROGRAM REQUIREMENTS

The 1994 auger sampling event was performed based on the crust burn issue DQO (Johnson 1994). The 1994 core sampling event was guided by the safety screening DQO (Babad and Redus 1994) and the flammability DQO (McDuffie and Johnson 1994). Implementation of the DQOs through tank characterization plans are summarized in Schreiber (1994a, 1994b, and 1995).

5.5.1 Safety Evaluation

Data criteria identified in the safety screening and flammability DQOs are used to assess the waste safety in tank 241-SY-103. The safety screening DQO requires data from two widely spaced risers, and the flammability DQO requires data from one riser. Because of the pressurization observed in the bottom segment for core 62, it was decided not to obtain a second core from the tank; therefore, the sampling requirements of the safety screening DQO (Babad and Redus 1994) were not met. Safety screening results for the vertical subsegments for the one core indicate the two major waste layers are relatively homogeneous. Because of the large amount of water in the tank and the gas evolution events, the waste may be mixed. It is possible that the horizontal variations that would be observed by taking and analyzing a second core would be small.

Although some of the auger samples of the crust had low moisture (< 17 weight percent) content and relatively high (1 weight percent) TOC concentrations, no exothermic reactions were observed. This indicates the potential for a crust burn is low.

Table 5-11 summarizes the results for the safety screening analyses. Most samples exhibited exothermic behavior. The mean enthalpy observed for a dried sample was approximately the same (350 to 400 J/g) for the supernate and solid phases of the waste. One drainable liquid and one solid semi-segment sample exceeded the present 480 J/g safety screening criteria. However, the weight percent water for the waste is significantly above 17 weight percent and would prevent propagation of any potential reaction.

The exothermic behavior is most likely the result of the reaction of organic complexants with nitrates/nitrites at elevated temperatures. Table 5-12 shows the TOC is relatively high throughout the tank. The samples with larger exotherms had dry weight TOC concentrations near 2 weight percent. All of the energy estimates (except two) calculated from the TOC, assuming that the TOC is acetate, were greater than the observed exotherms by DSC. These calculated enthalpy values are based on an estimate of 1,200 J/g energy for 4.5 weight percent TOC as acetate (Turner et al. 1995). Only small amounts of cyanide were found in the waste and do not contribute significantly to the observed exotherms.

Even though the sampling objective of two full-depth core samples was not met, subsequent evaluation has determined that the data for tank 241-SY-103 are sufficient to address the safety screening issue (Reynolds et al. 1999). Although some samples exhibited exotherms

exceeding the criteria, TOC analyses indicate that the fuel content of the waste is too low to propagate an energetic reaction. The safety screening issue is now considered closed for this tank.

Ion chromatography results for formate, acetate, and oxalate can account for 20 to 30 percent of the TOC in the supernate and 70 to 80 percent of the TOC in the solid phase. The solid phase contains significant quantities of oxalate, and the supernate contains none. This indicates that insoluble oxalates may be present in the solid waste. The oxalates and formates are degradation products of complexants such as HEDTA and EDTA and will not react as energetically with nitrate as the original complexants.

Radiolysis of water and organic degradation in the tank generate hydrogen and other gases (NH_3 , NO_x) in the headspace of the tank. Combustible gas meter testing of the tank vapors before sampling measured 0 percent LFL. The safety screening DQO notification limit for flammable gas concentration is 25 percent of the LFL (Dukelow et al. 1995). The combustible gas meter used to measure gases in the tank vapor reports results as a percent of the lower explosive limit (LEL). Because the National Fire Protection Association defines the term LFL and LEL identically, the two terms are used interchangeably (NFPA 1995).

Table 5-11. Comparison of Analytical Results with Decision Criteria for the Safety Screening Data Quality Objective.

Decision Variable	Decision Criteria Threshold	Analytical Values	
		Supernate Segment 1 to 9	Solids Segment 9 to 14
Total Fuel	-480 J/g ¹	mean = 384 + 53 J/g ²	mean = 347 + 67 J/g ²
		high = 559 J/g	high = 630 J/g
Percent Moisture	17 wt%	Av = 48.7 - 0.4 wt% ²	Av = 41.6 - 1.4 wt% ²
Total Alpha	1 g/L 61.5 μ Ci/mL Liquid 41.0 μ Ci/mL Solid	< 0.08 μ Ci/mL ³	Av = 0.95 + 0.15 μ Ci/mL ²
Flammable Gas	< 25% LFL	Explosivity meter = 0% LFL	
		Highest standard hydrogen monitoring system H ₂ = 7.35% LFL	
		Highest NH ₃ = 0.32% LFL	
TOC	30,000 μ g/g	9,640 μ g/mL (18,680) ⁴	10,600 μ g/g (15,820) ⁴

Notes:

¹Negative values denote exothermic reaction. The 480 J/g is based on the most recent version of the safety screening DQO (Dukelow et al 1995). A threshold of 523 J/g was applicable at the time of the sampling event.

²Upper or lower limit to a one-sided 95 percent confidence interval on the mean.

³Total alpha on the drainable liquid composite.

⁴Values in parentheses are based on dry weight.

Table 5-12. Evaluation of Organic Fuel Content in Tank 241-SY-103.

Sample	TOC	Oxalate	Formate	DSC Energy	Calc. Energy ¹
Strata/Segment	µg/g or mL	µg/g or mL	µg/g or mL	J/g Wet	J/g Wet
Strata A Segment 1-solids	4,770 (7,845)	< 95.8	2,920 (3,993)	118	127
Drainable liquid Comp. Segments 2 to 7	9,640 (18,680)	< 2,550 1,350 Acetate	4,240 (7,579)	85	257
Strata B Segments 4 to 8 solids	2,660 (4,990)	< 97.1	2,750 (5,159)	165	71
Segment 4 Lower solids	3,200 (4,526)	n/a	n/a	57	85
Segment 8 Drainable liquid	10,000 (19,157)	< 2,550 1330 Acetate	4,280 (7700)	227	267
Strata C Segment 9 solids	9,580 (17,514)	23,200 (42,413) 368 Acetate	3,440 (6289)	175	255
Segment 13 Lower solids	10,800 (18,060)	n/a	n/a	377	288
Segment 14 Lower solids	10,300 (17,487)	n/a	n/a	273	275
Strata D solids Comp.	10,600 (15,820)	20,800 (31,044) 3,130 Acetate	4,960 (7,420)	159	283

Notes:

() = Are dry weight values.

$$^1\text{Calculated Energy (J/g)} = \text{wt\% TOC in sample} \times \frac{1,200 \text{ J/g}}{4.5 \text{ wt\% TOC}}$$

A standard hydrogen monitoring system was installed on the tank in June 1994. An ammonia monitoring system also was installed on the stack exhaust for all SY tanks. For monitoring results, see Section 4.4. The highest recorded hydrogen concentration was 0.294 volume percent. This represents 7.35 percent of the LFL for hydrogen.

The estimated ammonia concentration from tank 241-SY-103 at the peak of the May 2, 1995 gas release was 486 ppmv. This represents only 0.32 percent of the LFL for ammonia. The standard hydrogen monitoring system hydrogen results have been verified by occasional grab samples. Small quantities of methane (10 to 15 ppmv) have also been detected in grab samples but do not contribute significantly to the LFL. This monitoring indicates the flammability of the tank vapors are well below the 25 percent LFL limit even during the short duration gas release events. Rheology, void fraction, and other physical measurements on the waste will be used to assess the potential for gas build-up in the liquid and solid phases of the wastes.

Another factor in assessing the tank waste safety is the heat generation and temperature of the wastes. Heat is generated in the tanks primarily from radioactive decay. The primary contributors for heat generation in the tank are ^{137}Cs and ^{90}Sr . The estimated heat generated from the isotopes in the tank is 5,880 W (20,100 Btu/hr) as shown in Table 5-13. This is well below the 11,723 W (40,000 Btu/hr) criteria for distinguishing a high heat tank from a low heat tank. Temperature monitoring indicates the waste temperature is decreasing as expected from decay of the isotopes.

Table 5-13. Heat Generation (W).

Matrix	$^{90}\text{Sr}/\text{Y}$	$^{137}\text{Cs}/\text{Ba}$	Total W
Drainable liquids	2.69E+1	2.70E+3	2.72E+3
Convective solids (stratum B)	1.25	1.19E+2	1.21E+2
Nonconvective solids (stratum D)	5.00E+2	2.54E+3	3.04E+3
			5.88E+3

The potential for criticality is assessed from total alpha and $^{239/240}\text{Pu}$ analyses. As expected, the highest total alpha results (0.5 to 1.5 uCi/g) were found in the solids layer. These results are well below the 41 uCi/g notification limit for safety screening. In addition, the $^{239/240}\text{Pu}$ activity in the solids is approximately 0.06 uCi/g. This and ^{241}Am analyses indicate that most total alpha activity is from ^{241}Am .

5.5.2 Operational Evaluations

The 1986 sampling was performed to characterize the waste for retrieval and processing to create immobile waste forms suitable for disposal. The 1994 core sampling was performed to screen the tank for general safety considerations, flammable gas issues, and further process development purposes. However, the process development core (core 2) has not been sampled yet. Metal and anion analyses will support operating decisions for this tank.

The 1994 analysis results indicate the total organic carbon content of the tank is near the 10-g/L TOC complexant waste classification limit, and the actinide levels in the sludge exceed the transuranics limit of 100 nCi/g.

5.5.3 Environmental Evaluation

Tank 241-SY-103 was not characterized to designate waste or to evaluate environmental compliance issues. The tank has been characterized to meet regulatory requirements that the waste is safely stored and managed. No specific organic (volatile or semivolatile) analyses have been performed on the tank; therefore, no assessment can be made of these compounds.

The 1994 analyses indicate the tank meets the hydroxide specification ($12 < \text{pH} < 14$), with the lowest pH measured at 12.85. Chromium, mostly as Cr^{3+} , is present in relatively high concentrations in the sludge. No analysis was made for metals such as lead, mercury, cadmium, and silver.

5.5.4 Process Development Evaluation

The metal and anion analyses will be important in evaluating the glass disposal waste formulations and identifying potential components that may affect the treatment and disposal process. Because waste sludges may be blended, washed, and treated before disposal, there are no specific criteria. Solids samples have been taken for physical testing (Bredt et al. 1995) and to evaluate sludge washing (Lumetta and Rapko 1995).

6.0 CONCLUSION AND RECOMMENDATIONS

The crust, supernate, and solids in tank 241-SY-103 were sampled and analyzed. Because no exotherms were observed in any auger crust sample, the potential for a crust burn was considered low, and full core sampling was performed. Only one core sample was taken in 1994 because sampling was stopped after a segment showed pressurization when extruded in the hot cells. The one core satisfied the flammability DQO but did not meet the full requirements of the safety screening DQO which requires two cores from widely spaced risers. Although sampling was not optimal, a recent evaluation has concluded that the sampling and analytical data for the tank are sufficient to address the safety screening DQO issue (Reynolds et al. 1999). Although some samples exhibited exotherms exceeding the criteria, TOC analyses indicate that the fuel content of the waste is too low to propagate an energetic reaction. The safety screening issue is now considered closed for this tank.

The DSC analyses for one drainable liquid and one semi-segment solid exceeded the safety screening criteria of 480 J/g (dry weight). All segments in the core exhibited exotherms. TOC levels were relatively high throughout the tank but less than 3 weight percent. The weight percent water concentration for samples was well above the 17 weight percent criteria; therefore, although a fuel source is present in the waste, the water content is too high for an exothermic reaction to propagate. The thermal history of the waste does not indicate excessive temperatures, and the tank temperature is decreasing.

Flammability testing of the tank vapor using a combustible gas meter before sampling indicated 0 percent of the LFL. Hydrogen gas monitors for tank 241-SY-103 have recorded hydrogen gas concentrations in the headspace as high as 7.35 percent of the LFL. Ammonia monitors on the SY Tank Farm stack exhaust have estimated ammonia concentrations of about 0.3 percent of the LFL during a gas release event. These values are consistent with results obtained from grab samples and are well below the 25 percent LFL vapor safety criteria. Based on these results, ignition of the tank vapors is not possible.

Physical measurements on samples from the 1994 sampling event and in-tank rheology and void space measurements have been made and will be used to evaluate gas accumulation in the tank waste. Analysis of metals, TOC, and anions further support the flammability DQO.

The total alpha results and the isotopic plutonium results show that the fissile content of the waste is well below the criticality criteria for the waste. The ^{241}Am concentration in the solids is about 10 times higher than the plutonium concentration and together they exceed the transuranic waste criteria of 100 nCi/g.

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